

CEMENT AND LIME MANUFACTURE

PUBLISHED ALTERNATE MONTHS.

PRICE 1/- A COPY.

ANNUAL SUBSCRIPTION 6s. POST FREE.

PUBLISHED BY
CONCRETE PUBLICATIONS LIMITED
14 DARTMOUTH STREET, LONDON, S.W.1

TELEPHONE: WHITEHALL 4561.
TELEGRAPHIC ADDRESS:
CONCRETUS, PARS, LONDON.

PUBLISHERS OF
"CONCRETE & CONSTRUCTIONAL ENGINEERING"
"CONCRETE BUILDING & CONCRETE PRODUCTS"
"CEMENT & LIME MANUFACTURE"
"THE CONCRETE YEAR BOOK"
"CONCRETE SERIES" BOOKS, ETC.

VOLUME XIX. NUMBER 5

SEPTEMBER 1946

Extraction of Alumina from Ashes.

A GERMAN PROCESS.

A REPORT has been published by H.M. Stationery Office (price 1s.) containing some notes by Dr. F. M. Lea on the Seailles-Dyckerhoff process of extracting alumina from coal ashes, colliery waste, and similar material. The description of the process has been written by Dr. Lea following a recent visit to the works of the Portlandzement Fabrik Dyckerhoff & Sohne at Amöneburg bei Biebrich.

The report states that two commercial plants to operate the process were established during the war. Though technically the process was found practicable it was quite uneconomical with materials of such low alumina content as those used. None of the pilot plant used in the early trials remained, and it was found that, after completion of the pilot plant trials at the Dyckerhoff works, commercial plants had been established at Rudersdorf (Berlin) and at Stromberg near Kitkowitz (Czechoslovakia), both using power station ashes as raw material.

Essentially the process consists of burning an alumina-containing raw material with limestone, extracting the pulverised material with water by a counter-current method, precipitating the alumina and lime from this solution by treatment with carbon dioxide, and subsequently extracting the alumina from the mixture of these two solids by treatment with soda. An alternative procedure is to treat the burned material with a sodium carbonate solution somewhat along the lines of the Pederson process, but using a lower sodium carbonate concentration.

In the pilot plant trials the Seailles water-extraction method was used, but this proved to be too expensive and was abandoned in favour of alkali extraction. The maximum alumina concentration obtained from the water extraction by the Seailles process was not more than 1 gramme Al_2O_3 per litre. In the alkali-extraction method the burned material was treated with a solution of 3 grammes Na_2CO_3 per litre at 40 to 50 deg. without sodium hydroxide. This gave 2 to 3 grammes of alumina per litre in the extract.

The object of establishing the plants was to produce alumina from indigenous raw materials. Both clay and colliery wastes from the Saare were tested, but eventually it was decided to use power station ashes with as high an alumina and as low a silica content as possible. A content of 30 to 35 per cent. alumina in the ash on a dry basis was desired, but material with a content of alumina between 25 and 30 per cent. was also apparently used. The mixture of ashes and limestone was treated as in Portland cement manufacture and burned in a rotary kiln with a reducing atmosphere to obtain the great bulk of the iron either as FeO or as metallic iron. The presence of ferric iron in the burnt material reduced the proportion of alumina that could be extracted. The temperature of burning was about 1300 deg. C. and the air flow through the cooler was reduced to give slow cooling and obtain a self-pulverising clinker. The lime content of the raw mix was calculated as that required to form $2\text{CaO} \cdot \text{SiO}_2$ and $\text{CaO} \cdot \text{Al}_2\text{O}_3$ with about 10 per cent. excess. No allowance was made in the proportioning for iron oxide. A clinker of this composition should be self-pulverising on slow cooling, and unless the clinker did self-pulverise it was found that the alumina content which could subsequently be extracted was reduced. For 1 ton of alumina, 10 to 12 tons of ashes (25 to 30 per cent. alumina) and 15 tons of CaCO_3 were required. The clinker contained 13 to 14 per cent. of alumina and from 60 to 70 per cent. of this could be extracted by the sodium carbonate method. The self-pulverised clinker was treated with sodium carbonate solution in a mixer from which both solution and solids passed to a sedimentation tank or thickener. The mud which settled out in the thickener was passed to rotary vacuum filters and then used for the manufacture of Portland cement. The solution from the thickener was passed through filter presses and then precipitated by treatment with carbon dioxide. For this purpose kiln gases were used, but this led to difficulty in plant operation and maintenance. The gases were first washed to remove dust and the wet gases containing sulphur dioxide caused trouble by corrosion of the pipelines. The solution obtained from the precipitator was returned to the circuit.

A typical analysis of the ashes used at the Stromberg plant was: Ignition loss, 8.8 per cent.; SiO_2 , 48.4 per cent.; Fe_2O_3 , 12.3 per cent.; Al_2O_3 , 28.75 per cent.; CaO , 0.50 per cent. Typical analyses of the clinkers were as follows:

	<i>Rudersdorf</i>		<i>Stromberg</i>
	(1)	(2)	
SiO_2	23.32	23.44	23.16
Fe_2O_3	5.35	5.35	3.20
Al_2O_3	13.49	11.24	13.72
CaO	54.66	54.94	55.95

The Stromberg clinker contains about 10 per cent. lime over that required for $2\text{CaO} \cdot \text{SiO}_2$ and $\text{CaO} \cdot \text{Al}_2\text{O}_3$, but the Rudersdorf clinkers contain 17 to 18 per cent. excess lime. It was stated that 10 per cent. excess was the correct value, but at Rudersdorf it was attempted for a time to work with the higher lime content. The ashes used for the Rudersdorf clinker were a mixture of ashes from Ruhr and Silesian coals obtained from the central power station in Berlin.

The ashes used at Stromberg came from Morava Ostrava. A high-grade limestone was needed, that at Stromberg containing about 97 per cent. CaCO_3 . The ferric oxide shown in these analyses is the total of all iron present as Fe, FeO, and Fe_2O_3 , calculated as Fe_2O_3 . The actual content of Fe_2O_3 was small. There was a fair amount of metallic Fe present as very small grains.

Though technically the process was found to be practicable, it was very expensive using raw materials so low in alumina content. At pre-war prices the cost of Bayer process alumina from high-grade bauxite was 120 to 150 marks per metric ton. The cost of material extracted from coal ashes as described was about 600 marks per ton. The cement plants used were old ones, and with more modern equipment it was thought that with the same raw materials it might have been possible to reduce the cost to 300 to 400 marks per ton, assuming a larger plant producing about 30,000 tons Al_2O_3 per year. The Stromberg plant was designed for 10,000 tons of alumina per year, but actually only produced about 6,000 tons. The Company concluded that the process is quite uneconomical with raw materials containing 30 per cent. alumina, and thought it would still be uneconomic if the raw material contained 40 per cent. of alumina, compared with the cost of alumina by the Bayer process using high-grade bauxites. It might, however, have more possibilities if applied to siliceous bauxites which contain too much silica for treatment by the Bayer process. A low-grade bauxite (40 per cent. Al_2O_3) was apparently tried at Stromberg, but was difficult to burn in a rotary kiln as serious clinker rings formed within 24 hours. Blastfurnace slag had not been tried as raw material, as it was stated that in general the German slags did not have above 15 to 16 per cent. Al_2O_3 content. The quality of the alumina obtained by the sodium carbonate extraction of the clinker was as good as was required for alumina for metallic aluminium production. In particular, the SiO_2 value was within the required limits.

The patents for precipitating the alumina from the aqueous calcium aluminate extract with carbon dioxide under pressure so as to avoid precipitation of the lime had never been used. The alumina precipitated along with calcium carbonate when the extract was precipitated by carbon dioxide was not readily soluble in sodium carbonate, and it had been found necessary to treat this precipitate with 10 to 15 per cent. sodium hydroxide solution at 100 deg. C. This was done by using sodium carbonate and causticizing it with lime. A small plant in Rumania produced 1 ton of Al_2O_3 per day from bauxite containing about 10 per cent. silica. There was no commercial plant in France, and only a small experimental plant in the Paris region. The residue from the alkali extraction process proved satisfactory for use in Portland cement manufacture and gave a good cement with a low alumina: iron-oxide ratio. The Dyckerhoff Company received a Government subsidy, which in part covered the losses on the development and operation of this process.

The Operation of Rotary Lime Kilns.

IN an article in "Rock Products" for February, 1946, Mr. V. J. Azbe states that a rotary kiln should produce a ton of lime per day per 35 cu. ft. of kiln volume; for example a 9 ft. by 175 ft. kiln should produce 250 tons of lime per day. Some of the factors that contribute to high efficiency are dealt with by the author, and are abstracted in the following.

Flow of Material.

The flow of material through a rotary kiln is proportional to its inclination, or rather more on the slope of the upper surface of material. Time of passage through the kiln is directly proportional to the speed of rotation, so excessive flow due to too great an inclination can be regulated by retarding the speed; it may also be regulated by varying the level by means of a choke. Time of passage is inversely proportional to the diameter of the kiln. For a 9-ft. kiln, the time would be 50 per cent. less than for a 6-ft. kiln for any given slope of material or speed of kiln. The rate of feed has no effect on the time of passage if the kiln is full, but the load carried and discharged is proportional to the rate of feed up to the point of rear over-flow. However, the rate of feed determines the back-end height of the stone, which in turn determines the slope of the material and therefore the amount of discharge.

The inlet end constriction should be the maximum possible without throttling the gases and thus limiting capacity. About 0.1 sq. ft. per ton of lime should be correct, but this depends very greatly on the draught available, and dust-chamber leakage. The outlet end should also be constricted consistent with practical limitations. Through this constriction the kiln load and kiln time are increased, wall surface exposure to heat is reduced, and lime surface increased. The feed should be constant and in direct proportion to the kiln rotation, so as to maintain a continuously full load in the kiln.

Capacity.

Rotary kiln capacity is mainly a factor of the amount of fuel that is properly burned. The amount of fuel that can be burned depends on the amount of air that passes through the kiln, which is a factor of kiln diameter and of draught. The draught that matters is that inside the kiln at the feed end and not that in the dust chamber. For maximum capacity it will be high at this end and lower at the discharge end. At low capacity this may be reversed. If the draught is high at the discharge end air is kept from entering the kiln freely, thus impairing capacity. If the draught is low at the feed end the kiln may be choked too much or the dust chamber may be plugged, or the dust chamber may be leaking, drawing in tramp air at the rear rather than useful air up in the kiln.

Up to a certain point increasing the capacity of the kiln also increases efficiency, although at any point with every increase the terminal temperature of the

gases will increase. Up to a certain limit the loss due to this is offset by a reduction in radiation loss, as this has a tendency to reduce with increase of capacity. Beyond this limit, however, efficiency will decrease with an increase of capacity. Most kilns have a relatively idle mid-section. Stone is rapidly preheated at one end and lime is made at the other end. Between these zones, temperature conditions are in an approximate equilibrium with little heat being transferred.

Heat Absorption and Efficiency.

When heat is developed in the kiln, its transfer to the lime takes place mostly by radiation from the hot flame. The hotter the flame or the cooler the lime surface the greater will be the heat transfer. Heat is also radiated to the kiln wall, but not as much per unit of surface as to the lime because the wall is hotter; but as there is more wall than lime surface, the proportion the wall receives is considerable. Some of the heat received by the wall will pass through the shell and be lost. Some will be re-radiated to the lime, but this flow is obstructed by the flame and a small amount will be passed to the lime by conduction when it rolls over the hot surface of the rotating kiln. Heat will also be given to both the lime and the wall by convection, but due to the very limited lime surface and wall surface exposed to the very large diameter stream of gas, there will be very little of this.

The heat transfer by radiation varies as the fourth power of the temperature difference, which is of great importance. For instance, if the hot radiating gas is 2,500 deg. F. and the lime surface 2,300 deg. F., 28,000 B.T.U. will be radiated per square foot. If the gas is still at 2,500 deg. F. but the lime surface is only at 2,200 deg. F., the radiation rate will increase to 40,000 B.T.U., an increase of 42 per cent. If the temperature of the gas is 2,000 deg. F. and that of the lime 1,800 deg. F., the radiation will be 15,500 B.T.U. If the lime temperature is dropped to 1,700 deg. F., for a 300 deg. F. difference radiation will be 22,500 B.T.U., an increase of 45 per cent.

We must not confuse the surface of the layer with the surface of the lime. The first is the corrugated surface of the layer of lime rolling in the kiln; the other is the outside surface of the individual pieces. This outside surface gathers heat which, due to the temperature differential, is conducted to the undissociated stone within the piece. The hotter the surface of the lime the greater the heat transfer will be. The "layer surface" may be quite hot while the "lime surface" may be comparatively cool. While the "layer surface" will always be hotter than "lime surface" throughout the mass, the "layer surface" should be as cool as possible in order to increase the radiant heat transfer, while keeping the "lime surface" as hot as possible to increase the heat transfer and so obtain quick conversion to lime. The lime on the surface is heated, and then it rolls under the surface and distributes the heat within itself.

In a slowly rotating kiln there is considerable time between exposures of lime to heat so that the "layer surface" becomes over hot, reducing its ability to

acquire heat, while, in the mass, temperatures equalize themselves, thus reducing lime-making action. This state can only be counteracted by high speed of rotation. If the gases are at 2,200 deg. F. and the lime at 2,000 deg. F., the gases would need to be raised to only 2,400 deg. F. to double the heat transfer if the lime remained at 2,000 deg. F. Flame temperature is important, and the higher the better within practical limits, but of even more importance is a low lime surface temperature. By this is not meant the average temperature of the lime at any point of the kiln cross-section, but rather that on the surface and nearest to the flame, as this lime is mainly receiving heat. According to the fourth power law, it needs to be but little lower in temperature to receive much more radiant heat from both the flame and kiln wall.

Lime surface temperature can be controlled only by speed of rotation, that is to bury the surface lime before it becomes heated too much and to make it reappear sooner. Then the surface will be cooler but the average temperature of the mass will be hotter, as the stone will be absorbing more heat. But speed controls the rate of discharge, and if the kiln be speeded without substantial outlet constriction it would soon be discharging core. It also would tend to drop the load and reduce the lime surface exposure and increase the wall exposure to heat, all of which are undesirable. The solution would appear to be a low slope of material for a high speed. However, the slope can also be varied by a combination of heavy discharge constriction, that would assure maintenance of hot zone load near to good normal width, then raising or lowering the stone level in accordance with the capacity possible. This unfortunately means a narrower stone layer at the feed end, but a wide one at the hot end, together with a lower temperature of upper surface for both lime and stone.

High Efficiency Operation.

Assume that the kiln gas temperature is 1,500 deg. F., and that the kiln is equipped with an efficient lime cooler, fully recovering sensible heat of the lime and returning all of it to the kiln. The heat necessary for calcination and preheating per pound of lime would then be: Calcination reaction at 1,500 deg. F., 1,220 B.T.U.; preheating of CO_2 portion of limestone, 390 B.T.U.; total heat abstracted from high elevation heat stream, 1,610 B.T.U. As all sensible heat of the lime is returned to the kiln none can be charged for, but since all of it came from the high intensity portion, there is no credit. The fuel ratio is increased but not to the extent possible when stone is preheated with waste heat. Suppose that all of the stone preheating is with heat of low elevation and all sensible heat in the lime is recovered and returned to the kiln. The total high heat requirement per pound of lime would then be: Calcination reaction at 1,500 deg. F., 1,220 B.T.U.; preheating of CaO portion of limestone (credit), 390 B.T.U.; total heat of high elevation, 830 B.T.U. Through preheating followed by cooling we recover low-intensity heat and recuperate it in the cooler to high intensity and thus accomplish a remarkable saving.

Summarizing the three conditions, we obtain :

	B.T.U. per lb. of lime	Fuel ratio.
High-heat stone preheating, no lime cooling . .	2,230	2.57
High-heat stone preheating, fully effective cooling . .	1,610	3.57
Low-heat stone preheating, fully effective cooling . .	830	6.92

Temperature Conditions in a Rotary Kiln.

In the operation of rotary kilns we know what the gas, lime or stone temperature, or the gas analysis, is at its approximate terminals. Not much, however, is known of the regions between these points nor of the temperatures prevailing in the mass of rotating material. In respect to lime or stone on the slope of the segment, the heaviest pieces tend to roll the farthest and segregation occurs, and thereafter the mass tends to roll concentrically. The hottest lime or stone from the surface tends to roll under and into contact with the hot wall and in time reappears at the top edge of the segment to become again exposed to the radiant heat of the flaming gases. Thus the largest or heaviest portion of the mass is favoured at the expense of the lighter portions, which remain buried deeper within the mass, being dependent practically entirely on heat derived from adjacent lime. As long as the sizes are in a limited range, there is a replacement of the portion rotating on the outside of the mass when due to calcination it becomes unbalanced in weight with the layers underneath, which are heavier and contain more core. If the size range is too great, however, this cannot take place. Thus there is a great range of temperature through the cross-section of any part of the kiln and, while the top surface of the lime may be very hot, deep in the mass the available heat is taken up so rapidly by the high-core-containing portions that temperatures are barely of calcination intensity. This must be so or the tendency for the smallest pieces to have most core would not be so pronounced.

Similarly puzzling conditions apply to combustion—how far does it extend and what are the comparative terminal conditions at the end of the calcining zone and at the end of the kiln? Stone at the feed end of the kiln heats very rapidly because the temperature difference between the stone and the gas is very high. One would assume that rapid heating of the stone would also call for rapid cooling of the gas, but the gas cools slowly. While the heating rate of stone is represented by a curved line, the rate for gas is represented by virtually a straight line. The flow through a test kiln was found to be about 4 lb. of gas per lb. of lime or for each 1.78 lb. of stone. Considering these weights in conjunction with their respective specific heats, then at a fuel ratio of 3.5 to 1 the gases should cool by approximately half the amount the stone preheats. Actually, however, the gas cooling rate is not near this figure and may not be more than a fourth of the stone range. Tests indicate that calcination begins at about 60 to 80 ft. from the feed end. The gas temperature in this region is between 2,000 deg. F. and 2,200 deg. F., but, if we assume a temperature of only 2,000 deg. F. for gas and 1,600 deg. F. for stone

again we have the discrepancy of too slow cooling of the gas for the amount of stone passing through. The reason for this could only be delayed combustion (the burning of gases in the stone preheating zone), thus supplying by combustion some of the heat of preheating and preventing the gases from cooling as one would expect.

Often kilns flame into the dust chamber. This occurs ordinarily because an insufficient amount of air comes through the kiln for complete combustion and, on contact with leakage air entering at this point, secondary combustion takes place. Delayed combustion is a different matter; air is present, but mixing is poor, so combustion may extend far into the stone preheating zone, as was demonstrated during tests. At one testing station, 60 ft. from the feed end of the kiln, gases were withdrawn from different cross-sections. One sample taken a good distance from the stone surface had a content of 21 per cent. CO_2 , 2 per cent. O, and 4 per cent. CO. Another taken close to the stone had a content of 32.2 per cent. CO_2 , 1.2 per cent. O, and 3.4 per cent. CO. The great difference in CO_2 content of the two samples shows how stratified is the flow of gases and reveals also that the cooled gases along the stone and lime surface have no tendency to move up and be replaced by hotter streams. In this case the loss into the preheating zone of combustible gases, sometimes called loss due to incomplete combustion, is very great, as 4 per cent. CO, when diluted with air and CO_2 from the stone, is close to 5 per cent. CO in straight combustion gases, which in turn means a heat loss equivalent to about 25 per cent. of that initially available in the carbon. No data were obtained on the temperature prevailing through the layer of lime and stone.

The fact that the surface temperature of lime in the idle zone was 2,000 deg. F., although calcination had just begun, was because the cool stone was not brought to the surface and there was an idle zone where heat interchange should be high. Sluggish combustion was also a factor. Twenty-five feet from the discharge end of the kiln the temperatures of the lime surface and of the gas were at the highest point, although calcination was only 80 per cent. complete. Gas temperature was very high (2,950 deg. F.), and lime temperature was 2,500 deg. F. With lime at such a high temperature one would expect it to be completely burned, but calcination was only about 80 per cent. complete, again indicating the great difference which must prevail between the lime surface exposed to the gases or touching the hot wall and the general temperature of that removed from locations of highest heat. The rate of penetration of the zone of calcination into an individual piece of stone at 2,500 deg. F. is so rapid that in any size of rotary kiln the existence of lime core would not be imagined. Even at 2,300 deg. F. the rate of penetration is 2 in. per hour, so any 1-in. piece would become lime in 15 minutes. If it is not lime it has not been heated to this temperature at all, or not for a sufficient length of time.

Between the hottest point of the kiln and its discharge end, a further peculiar condition exists. While gas temperature is rising very fast, lime temperature is dropping, even after calcination is complete. Since calcination is complete, or

almost so, the temperature should remain high. In explanation of this we may assume that air delivered through the fuel pipe, combined with the combustible, constitutes the flame. Its temperature was measured. Surrounding this inner core of the gas stream is the secondary air entering the kiln, either through the cooler, the seal ring, or the many openings in the front of the hood. This secondary air cools the surface of the lime appearing at the upper surface and this in turn cools the rest of the lime in the mass. If more air entered as primary air through the burner, lime would stay hotter longer. If almost all could enter in this way, it would remain at the heat to which it was brought. To cool lime in the kiln is economically unsound; lime should be cooled in the cooler. If more air entered as primary air, or if secondary air entered closely adjacent to the primary stream, the temperature peak would not be as high and would be longer. It is probable that the hottest point would be within the fireman's range of vision, which is not so now. As it is, the primary stream enters at high turbulence, being slowed immediately by the secondary stream. Entering together, they could be given a rotary action which would carry far down through the kiln and tend to remove cooled gases from the lime surface and replace them with hot gases. Combustion would also be complete earlier.

This study indicates that, of the handicaps the rotary kiln is subject to, mixing of gases, lime and stone is so poor that the kiln cannot take advantage of heat transfer by convection and is dependent only on radiation and conduction. Mixing improves any heating or combustion apparatus. A dam at the discharge end helps in building up the load and its surface and time of travel, but it does not help mixing. Other chokes through the kiln would, however, not only build up the load for other stretches but would also break up stone stratifications and would tend to mix the gases through this baffling effect, tending to more complete earlier combustion and mixing of the cool strata adjacent to the lime with the very hot strata from the upper kiln cross-section. Such chokes would also function as curtains, in a measure shielding the hot zone from excessive loss of heat by radiation into the cooler sections of the kiln. They should help to provide the loss of draught through additional flow resistance.

Export of British Cement Plant.

Among the orders Messrs. Vickers-Armstrongs, Ltd., have in hand for cement-making plant for export are (a) The machinery for a complete cement plant for the Middle East, with a capacity of 250,000 tons per annum; (b) The machinery for a complete cement plant having a capacity of 100,000 tons per annum for Ceylon; (c) Machinery, including rotary kilns and mills, for the Far East; (d) Four rotary kilns and coolers, 4 raw mill grinding plants, and crushing plant for extensions to various works in South Africa; (e) A wash-mill plant for France; (f) rotary kilns for Mexico; and (g) Rotary kilns and mills for India.

Progress of the British Cement Industry.

ON his appointment as President of the Associated Portland Cement Manufacturers, Ltd., of which Company he had been Chairman for twenty-two years, Sir P. Malcolm Stewart, Bart., gave an interesting account of the development of the industry during the past twenty years or so, including some notes on the remarkable increase in the efficiency of manufacturing processes. The following is an abstract of his address, which was given at the last annual general meeting of the Company.

In 1924 a policy of considerable expansion involving much scrapping of plant, some of which was but partially obsolete, was adopted. Bevens works, situated on the Thames, offered the maximum of economic opportunity and was tackled first. Twelve rotary kilns producing 5,000 tons per week were scrapped and replaced by four kilns with a combined output of 10,000 tons per week. The resultant success amply justified the heavy outlay. Facilities were provided for export and coastwise trade on a large scale. The new wharf and jetty, costing over £100,000, made it possible to load larger coastwise vessels and to permit of liners coming alongside at all states of the tide. The new Bevens works was the prototype of many reconstructions and extensions on the Thames and Medway and at inland works, which in turn were brought to a high state of efficiency. Works rendered redundant through the creation of increased capacity were maintained in working order so that their output could be quickly obtained should unlooked-for demand arise. The programme was carried out over a period of years, continuously increasing productive capacity to keep it well ahead of any prospective demand. Perhaps one of the most striking aspects of the attainment of increased efficiency is that it often brings in its train relief from the most arduous features of manual work and a diminution of risk of accident, factors which the well-balanced designer should always bear in mind. Lime works were similarly dealt with. The cost of production per ton of cement at the works between the years 1925 and 1939 was reduced by 32 per cent. Whereas £100 locked up in fixed assets in 1925 represented 29 tons of annual productive capacity, to-day the figure is in the neighbourhood of 126 tons. In 1924 the weekly production per man was $6\frac{1}{2}$ tons. In 1939 it was $18\frac{1}{2}$ tons, a figure which equals that obtained from the average efficiency of the U.S.A. cement industry in that year. The expansion of productive capacity went far towards absorbing the displaced labour. The continuity of such satisfactory results can only be looked for in the future provided there is freedom for the exercise of unrestricted responsibility and enterprise, and for the taking of risks. This country can never regain industrial supremacy whilst we are cramped in action and daily delayed in the time-wasting task of the filling up of endless forms, many of which, when completed, have to be studied by officials, some of whom have not had the practical experience of industry necessary to interpret them. Both business and domestic life are made unbearably complex, whereas one of the arts of good management is to secure the maximum of simplicity of procedure.

Thermal Analysis of Clay.

THE United States Bureau of Mines has recently issued a number of papers dealing with the thermal analysis of clays and other aluminous materials. In "Technical Paper 644" (1945), an apparatus for differential thermal analysis is described. The mineralogical composition of a clay determined by these methods can be correlated with the alumina extractable with dilute sulphuric acid after calcination of the sample for one hour at 800 deg. C. The alumina of kaolinite and halloysite is virtually completely soluble, but that of dickite is not. The alumina content of montmorillonite and illite is only 20 and 25 per cent. respectively. Only 20 to 30 per cent. of the alumina content of montmorillonite and 50 per cent. of the alumina content of illite is extractable. Gibbsite in the clay raises the amount of alumina but lowers the amount extractable to 43 per cent. The extractable alumina in diasporite and boehmite is still less. Extraction from uncalcined samples is in general unsatisfactory. The kaolin and beidellite groups have the maximum solubility when calcined at temperatures between their strongest endothermic and exothermic reactions, illites at temperatures between their second and third endothermic reactions, and montmorillonites in the uncalcined condition. It is shown that there was good extraction of alumina and low extraction of silica in pressure digestion.

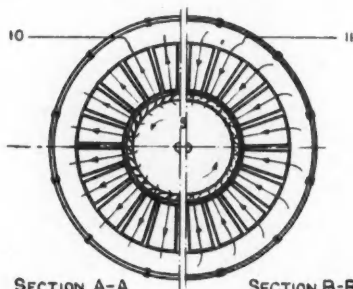
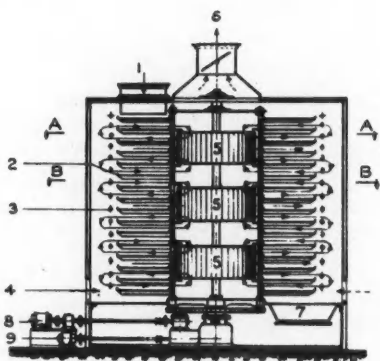
In "Report of Investigation 3764" (1944) the limitations as well as the applications of thermal analysis are investigated. The temperature at which thermal reactions take place and the magnitude of these effects have been determined by the following method. A specimen of the clay and a specimen of a thermally inert substance are electrically heated in a nickel block at a constant rate of 12 deg. C. per minute and the temperature differences between the two specimens are recorded by means of a differential thermocouple. From the observations a series of thermal curves for each sample is analysed and compared with those obtained by other workers. The thermal analysis method is considered to be quantitatively applicable within ± 5 per cent. to mixtures the main constituents of which can only be analysed individually to establish standard peak exothermic or endothermic areas. It is considered as applicable semi-quantitatively within ± 10 per cent. or qualitatively to aluminosilicate minerals, although confirmation by petrographic and X-ray methods is here necessary. Diasporite and kaolinite are easily distinguishable from gibbsite by this method, but the montmorillonite peaks differ in position or area, thus making quantitative determination of this mineral in a mixture uncertain. Decreased particle size or increased surface area in kaolin decrease the reaction energy associated with the differential thermal peaks and lowers the temperature at which endothermic decomposition of kaolin occurs. The clays examined varied widely in composition ranging from pure kaolinite through admixtures of kaolinite with gibbsite, montmorillonite, and illite to fairly pure three-layer lattice minerals. Commercial kaolins and ball clays were found to be predominantly kaolinite with some organic matter as shown by an exothermic peak. The initial 150 deg. C.

endothermic peak becomes more marked as the particle size decreases owing to the larger amount of adsorbed water on the increased surface.

The preceding abstracts are based on references given in "Building Science Abstracts," May, 1945.

A German Coal Dryer.

A REPORT by Mr. H. Bardgett (Ministry of Fuel and Power) and Messrs. F. H. Reed and L. D. Schmidt (United States Bureau of Mines) on a new type of coal dryer manufactured by Büttner-Werke A.G., of Uerdingen-Krefeld, has been issued by H.M. Stationery Office (price 1s.). The machine (*Fig. 1*) is of the turbine type. The coal is fed in at the top and passes downwards on a series of



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|--------------------------|----------------------|----------------------|
| 1. Wet coal inlet. | 5. Turbines. | 9. Turbine drive. |
| 2. Ring-disc frame work. | 6. Waste air outlet. | 10. Forward current. |
| 3. Heating coils. | 7. Dry coal outlet. | 11. Return current. |
| 4. Air inlet. | 8. Ring disc drive. | |

Fig. 1.

horizontal metal discs mounted in a vertical cylindrical metal shell in such a manner that they can be rotated. Each disc has a series of radial slots through which the coal is transferred to the disc below by fixed scraper plates. As the discs rotate, saw-toothed levelling arms spread the coal to a uniform depth on the discs. The discs are rotated about one revolution in three minutes, but the speed can be varied depending on the moisture content of the coal. Mounted on a central vertical shaft are three centrifugal fans, or turbines, rotated at about 6 ft. 6 in. per second, for circulating heated air or gases over the coal on the discs. In one type of dryer heating of the air is effected by steam pipes mounted vertically near the outer edges of the discs. In other types the heating is by the products of combustion of gas or solid fuel burned in an external combustion chamber.

The dimensions of commercial driers of this type are approximately as follows: Height 33 ft.; diameter of discs, 25 ft.; diameter of turbines, 15 ft.; capacity, 50 to 60 tons per hour. The maximum temperature in the steam-heated plants is 120 to 130 deg. C., and in those heated by combustion gases it is normally 220 to 250 deg. C. The company claims that the temperature of the dried coal can be controlled to within 2 deg. C. In plants heated by gas and by solid fuel, the oxygen contents of the waste gases are about 5 per cent. and 8 per cent. respectively.

Dryers have been designed for the treatment of coal of different sizes. The firm claims that an important feature is that there is no loss of dust and no auxiliary de-dusting apparatus is necessary. This is due to the low velocity of drying gases through the plant. It is also claimed that there is no appreciable alteration of the size of the coal during drying. The heat requirements are 1,980 B.Th.U. per lb. of water evaporated. The power requirements are said to be low.

United States Specifications for Portland Cements.

A NUMBER of revisions of the American standard specifications for Portland cements were approved by the American Society for Testing Materials in June last.

The SO_3 content of ordinary Portland cement is raised from 2 per cent. to 2.5 per cent. when the C_3A content is greater than 8 per cent. In the case of high-early-strength Portland cement the SO_3 content is raised from 2.5 per cent. to 3 per cent. when the C_3A content is greater than 8 per cent.

Tentative specifications for air-entraining Portland cement (C 175-46T) were modified to delete the words restricting this cement for use in roads: to raise the permissible limits of air content of the standard mortar from 14 per cent. to 16 per cent.; and to set the maximum limit for SO_3 content at 2.5 per cent. when the C_3A content is greater than 8 per cent.

The Determination of Titania in Portland Cement.

A METHOD of determining the content of titania in Portland cement, by which six tests can be made in a day to an accuracy of 0.003 per cent., is described by Mr. J. J. Diamond, of the United States National Bureau of Standards, in "Rock Products" for April 1946. The following is an abstract of the article.

Of the methods available, the colorimetric method involving the use of hydrogen peroxide has been selected because of its simplicity and adaptability. The method involves the isolation of the R_2O_3 , its fusion with $K_2S_2O_7$, the solution of the melt in 1 : 9 H_2SO_4 , the addition of hydrogen peroxide to an aliquot to develop the peroxidised titanium colour and the measurement at the wave length of maximum absorption of the colour intensity using a Coleman Universal Spectrophotometer, and the compensating blank method. The preliminary precipitation of the titania with the R_2O_3 was adopted for several reasons. First, the R_2O_3 is often determined in the routine analysis of cement; secondly, the method can be applied to most silicate and carbonate materials since the R_2O_3 obtained from them is similar in character to that obtained from cement. A photoelectric instrument is used in preference to a visual comparator because experience has shown that personal factors severely limit the accuracy of the latter, even in the hands of those with moderate experience in its use. Using the proposed procedure, the average analyst can isolate the R_2O_3 from six samples of cement in a day. The determination of their TiO_2 contents can then be completed in three or four hours.

APPARATUS.—The instrument used in the proposed method is a Coleman Universal Spectrophotometer, Model 11, having a fixed slit width of 35 millimicrons. Essentially monochromatic light is produced by a grating and auxiliary filter, and the light transmitted by a solution is measured photoelectrically. The instrument is used with matched square cuvettes having a light path of 13 mm. and an 8-volt storage battery as a power source. A 40-ml. pipette should be used. This size permits two aliquots to be taken from a 100-ml. volumetric flask, leaving enough extra solution for waste and for rinsing the pipette. Each aliquot is sufficiently large to permit rinsing the cuvettes thoroughly and filling them at least half full for the reading.

REAGENTS.—Use the 30 per cent. hydrogen peroxide solution and keep it refrigerated to minimise deterioration. Use National Bureau of Standards Sample No. 154, Titanium dioxide (98.7 per cent. TiO_2) and make up a standard solution containing about 1 mg. per ml. by heating a suitable amount with an $(NH_4)_2SO_4$ and H_2SO_4 mixture, as described in the Provisional Certificate of Analysis accompanying the standard sample.

Procedure.

SEPARATION OF R_2O_3 .—Using a stirring rod, thoroughly grind together 0.5 g. cement and 0.5 g. NH_4Cl in a 50-ml. beaker. Cover with a watch glass and carefully add 5 ml. concentrated HCl. Add two drops concentrated HNO_3 ,

and stir to aid decomposition. Heat on the steam bath for about 30 minutes and stir occasionally to get complete decomposition of the cement. Filter through a 9 cm. medium-retention filter paper, wash ten times with hot 1 : 99 HCl and five times with hot water. Burn off the paper in a platinum crucible and treat the silica with 5 to 10 ml. HF and a few drops of 1 : 1 H_2SO_4 . Fume off the silica, HF and H_2SO_4 completely. Reserve the HF residue. Bring the filtrate from the silica to a boil, add methyl red indicator, and precipitate the R_2O_3 with NH_4OH . Filter hot through a 9 cm. medium retention filter paper and wash ten times with hot 2 per cent. NH_4Cl solution (slightly alkaline to methyl red). Burn off the paper in the crucible containing the HF residue from the silica.

$K_2S_2O_7$ Fusion (Recommended).—To the R_2O_3 obtained, or by any other procedure, add 5 g. $K_2S_2O_7$ and fuse over a low flame until solution is complete. Cool, separate the melt from the crucible, and transfer it to the beaker in which the R_2O_3 was precipitated. Wash the crucible and lid with hot water, using a

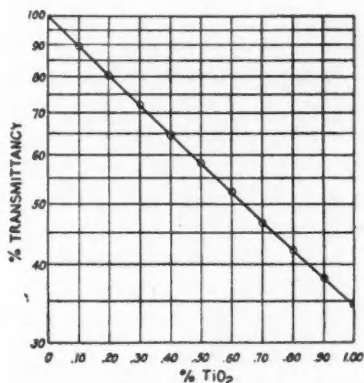


Fig. 1.

policeman, and add the washings to the beaker. Add 10 ml. concentrated H_2SO_4 and some water, cover, and digest on the steam bath with occasional stirring until the solution is clear. Cool to room temperature, pour into a 100-ml. volumetric flask and dilute to the mark with water.

Na_2CO_3 Fusion (Optional).—If the presence of interfering amounts of V, Mo, P or As is suspected, the following procedure should be used. Add 5 g. Na_2CO_3 to the R_2O_3 , fuse for 10 to 15 minutes, cool, separate from the crucible and transfer to a small beaker, and wash the crucible with hot water, using a policeman. Digest the melt and washings until the melt is completely disintegrated. Then filter through a 9 cm. medium retention filter paper and wash a few times with 2 per cent. Na_2CO_3 solution. Discard the filtrate, burn the paper and residue in the platinum crucible, fuse with 5 g. $K_2S_2O_7$, and then proceed as previously recommended.

COLORIMETRIC COMPARISON (Recommended).—Pipette two 40-ml. aliquots into clean dry beakers. From small burettes add 0.5 ml. 30 per cent. H_2O_2 to one and 0.5 ml. H_2O to the other. Mix each well. Rinse one cuvette several times with the coloured solution and fill it about half full. Use the reference solution similarly to rinse and fill the other cuvette. Wipe the outside of the cuvettes perfectly clean and dry before placing them in the cuvette holder. The same cuvette should always be used for the reference solution, the cuvettes should always be oriented in the same direction, and they should not be interchanged in the cuvette carrier. Place the unperoxidised "compensating blank" solution in the light path and set the instrument to 100 per cent. transmittancy at a wave length of 410 millimicrons. Then substitute the peroxidised solution and observe the transmittancy to 0.1 per cent. Read the TiO_2 equivalent from the calibration curve and divide by the sample weight to get the percentage of titania.

CALIBRATION.—Make up a standard titanium solution containing about 1 mg. per ml. of TiO_2 , as described under "Reagents." Using a burette, add accurately measured volumes of standard titanium solution to ten 100-ml. volumetric flasks to give a series of standards containing 0.5 to 5 mg. in increments of 0.5 mg. Add to each a solution containing 5 g. $\text{K}_2\text{S}_2\text{O}_7$ and 10 ml. concentrated H_2SO_4 and dilute to the mark with water. Take two 40-ml. aliquots of each solution, add 0.5 ml. 30 per cent. H_2O_2 to one and 0.5 ml. water to the other. Observe the transmittancy of the coloured solution using the colourless solution as a reference. Multiple determinations should be made for greater precision. Plot on semi-logarithmic paper to obtain a graph similar to that shown in *Fig. 1*; the curve obtained is almost but not quite a straight line.

COLORIMETRIC COMPARISON (Optional).—Place a mark on the 100-ml. volumetric flask at the 70-ml. level. After the sample has been diluted to the 100-ml. mark, use exactly 30 ml. to rinse the reference cuvette and fill it with solution, pouring off any of this 30 ml. which is not used. To the 70 ml. remaining in the flask add 1 ml. 30 per cent. hydrogen peroxide and rinse and fill the other cuvette with the resulting solution. Observe the percentage of transmittancy as described, and read the percentage of TiO_2 from a curve constructed using this optical procedure. The method is nearly as precise as the recommended procedure. Since the colour due to the iron, etc., is diluted in the peroxidised solution to 70 per cent. of its value in the reference solution, a negative error in the TiO_2 value results. The error varies with the amount of iron and other colouring materials present, and corresponds to about -0.002 per cent. for a cement containing about 5 per cent. Fe_2O_3 . The advantage of this procedure is its great speed and simplicity.

Discussion.

If present in a cement analysed according to the recommended procedure, V and Mo would react with H_2O_2 to give coloured complexes; and P and As

would tend to bleach the Ti complex. From the results shown in Table I, of

Table I

Cement	Flux	Number of determinations	Per cent. TiO_2			Average deviation
			Max.	Min.	Mean	
1	Na_2CO_3	3	0.164	0.160	0.163	0.0017
	$\text{K}_2\text{S}_2\text{O}_7$	3	0.170	0.164	0.167	0.0023
2	Na_2CO_3	3	0.187	0.180	0.184	0.0027
	$\text{K}_2\text{S}_2\text{O}_7$	6	0.191	0.184	0.188	0.0025
3	Na_2CO_3	3	0.250	0.241	0.245	0.0030
	$\text{K}_2\text{S}_2\text{O}_7$	3	0.251	0.248	0.249	0.0013
4	$\text{K}_2\text{S}_2\text{O}_7$	5	0.174	0.166	0.168	0.0020
	$\text{K}_2\text{S}_2\text{O}_7$	3	0.205	0.205	0.205	0.0000
6	$\text{K}_2\text{S}_2\text{O}_7$	5	0.250	0.245	0.246	0.0014
	$\text{K}_2\text{S}_2\text{O}_7$	5	0.338	0.327	0.333	0.0028

parallel analyses of typical cements, involving fusion of the R_2O_3 with Na_2CO_3 and with $\text{K}_2\text{S}_2\text{O}_7$, it is evident that there is normally little or no interference from these sources. The alternative procedure has been provided for use when the presence of significant quantities of P, As, V or Mo is suspected. The compensating blank technique has been incorporated to eliminate the interference due to iron and other coloured ions that accompany the R_2O_3 and the colour of whose solutions is not affected by the addition of hydrogen peroxide. Most ordinary Portland cements contain about 2 to 6 per cent. of Fe_2O_3 .

EFFECT OF REAGENT CONCENTRATION.—Lange states that one ml. of 30 per cent. H_2O_2 added to 100 ml. of solution will give maximum colour development up to the solubility point of the titanium complex. An excess of peroxide does not affect the colour. Experiment has shown that for an average Portland cement containing about 0.25 per cent. TiO_2 , a minimum of 0.2 to 0.3 ml. of 30 per cent. H_2O_2 per 100 ml. of solution is required for full colour development. An excess of peroxide is used in the proposed procedure to guard against incomplete colour development due to deterioration of the reagent.

REACTION TIME.—Trials have demonstrated that the formation of the coloured titanium complex is completed practically instantaneously. Maximum colour development was never found to take more than the minute or two required for manipulation before a reading could be taken. Ayres and Vienneau have found that the colour is stable, within the limit of error of the colorimeter they used, for a period of two years.

PRECISION AND ACCURACY.—The proposed method gives results which are precise (see Table I). To get a measure of the accuracy of the proposed method, National Bureau of Standards Standard Samples Nos. 102, Silica Brick, and 1A₇ Argillaceous Limestone, were analysed for titania. Suitable solutions of the titania were obtained by fusion of the sample with Na_2CO_3 , solution in HCl, one dehydration and filtration of the silica, precipitation of the R_2O_3 and fusion of the R_2O_3 and HF residue from the silica with $\text{K}_2\text{S}_2\text{O}_7$. Thereafter the recommended procedure was used.

Titania percentages of 0.158 and 0.158 were obtained in duplicate analyses of the silica brick, which is certified to contain 0.16 per cent. Titania percentages

of 0.167 and 0.170 were obtained in duplicate analyses of the argillaceous limestone, which is certified to contain 0.16 per cent. The results by different analysts averaged to obtain the latter value were 0.11, 0.16, 0.16, 0.17, 0.18 and 0.20 per cent.

TYPICAL ANALYSES.—Analyses of about 25 typical Portland cements of American manufacture, including the standard, low and moderate heat-of-hardening, and high-early-strength types showed titania contents ranging from 0.17 per cent. to 0.33 per cent. A natural cement and an aluminous cement were found to contain 0.24 per cent. and 1.31 per cent. respectively.

The Effect of Gypsum on Portland Cement.

In order to obtain comment, a paper entitled "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes," by William Lerch, and which is to be published in Vol. 46 of the Proceedings of the American Society for Testing Materials, has been published in advance by the American Portland Cement Association. It is pointed out that the paper is subject to modification.

In a synopsis, the author states that the manner in which the added gypsum controls the setting of cement has been the subject of many investigations and of some controversy. It is generally agreed, however, that its effect is to stop the rapid reaction normally shown by the calcium aluminates. In the absence of precise information relative to the function of gypsum in regulating the initial hydration, and in order to provide ample protection against the abnormal expansion that might result from the use of excessive amounts of gypsum, it has been the custom to place a limit on the SO_3 content of Portland cement in standard specifications. In the A.S.T.M. specification this limit has not been changed since 1917 except to permit a higher value for high-early-strength cement. In the belief that the quantity of gypsum required to regulate properly the hydration and hardening would vary with cement composition and fineness, the studies described in this paper were undertaken.

Twelve commercial clinkers covering the range of chemical composition generally encountered in Portland cements were selected for study. Each composition was used with various SO_3 contents, with uniform fineness of about 1,900 sq. cm. per gramme. In addition, five of the clinkers were ground to varying degrees of fineness with SO_3 constant at about 1.8 per cent. The effect of SO_3 on the rate of hydration was measured with a conduction calorimeter on neat cement pastes. Mortar prisms were used for physical tests. The studies have shown that a proper regulation of the reaction of early hardening is of much greater importance than merely regulating the time of setting. Such regulation

not only influences the rate of hydration, but affects the strength and the volume-change characteristics of the hardened cement paste. With some cement compositions the gypsum retards the initial hydration and set, while with others it acts as an accelerator. For many cements the strengths can be increased and the contraction on drying decreased by the use of larger additions of gypsum than are permitted by current specifications. The quantity required to obtain the best strength and the least contraction can be used without danger of abnormal expansion in water storage. The results show that the fineness, the alkalis, and the $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content all influence the gypsum requirements. The paper contains a discussion of the function of gypsum in regulating the reactions of hydration and hardening.

The results of the tests are summarised as follows :

A properly retarded cement is defined on the basis of the shape of the heat-liberation curve during the first 30 hours of hydration. A properly retarded cement is one that contains the minimum quantity of gypsum required to give a heat-liberation curve that shows two cycles of ascending and descending rates and that shows no appreciable change with larger additions of gypsum. The results of the physical tests show that, when considering cements ground from a given clinker, those containing the proper amount of gypsum to give this type of curve will develop the highest strength and the lowest contraction.

With cements of low alkali content those of high $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content require larger additions of gypsum than those of low $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content. For cements of the same $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content those high in alkalis react with gypsum more rapidly and require larger additions of gypsum than those low in alkalis. There is some evidence that cements containing Na_2O require larger amounts of gypsum than do similar cements containing an equivalent quantity of K_2O . Gypsum retards the early hydration of cements of high or moderately high $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content and accelerates the hydration of cements of low $3\text{CaO}\cdot\text{Al}_2\text{O}_3$.

Without added gypsum, cements of high or moderately high $\text{CaO}\cdot\text{Al}_2\text{O}_3$ content regardless of their alkali content, and cements of low $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ that are high in alkalis, react with water so rapidly that an immediate set occurs. This is the result of the rapid solution of the anhydrous aluminate phases and the rapid crystallization of hydrated calcium aluminates. With added gypsum, the gypsum dissolves in the mixing water together with lime formed by the hydrolysis of compounds present in the cement. The saturated lime-gypsum solution depresses the solubility of alumina in the aqueous solution and thereby retards the hydration of the aluminate phases.

In the presence of the saturated lime-gypsum solution the aluminate phases dissolve at a retarded rate and react with the lime and gypsum to form an insoluble calcium sulphoaluminate. By this process the gypsum may eventually become depleted and its concentration in the aqueous solution decreased. If at this time there is still present a quantity of unhydrated aluminate phases, a rapid reaction will occur. Such a rapid reaction appears to be the same as that which occurs in the immediate flash-set, that is, a rapid solution of the anhydrous

aluminate-phases and a rapid crystallization of hydrated calcium aluminates. With larger additions of gypsum this rapid reaction is eliminated and larger amounts of calcium sulphoaluminate are formed in place of hydrated calcium aluminates.

It appears that at least part of the alkalis of the cement are present in the aluminate phases, and that aluminate phases containing alkalis react with water more rapidly than do similar phases which are alkali-free or of lower alkali content. Thus cements with higher alkali content require larger additions of gypsum for proper retardation than do similar cements of lower alkali content.

Increasing the specific surface of the cement increases the quantity of aluminate phases available for reaction with water at early ages and thereby increases the quantity of gypsum required for proper retardation of cements of moderately high or high $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content.

Cements of low $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ and low alkali content without added gypsum can be mixed with water without the occurrence of a flash set. With cements of this type an amorphous hydrated calcium ferrite precipitates on the surface of the cement particles and seals the surface in a manner such as to retard subsequent hydration. With added gypsum a crystalline hydrated calcium sulpho-ferrite is formed which does not seal the surface and the hydration is accelerated.

The results show that varying the gypsum content of the cement will alter the rate of hydration products that are formed. It seems probable that such changes in the hydration process would alter the structure of the hardened paste. No attempt was made to examine the structure of the pastes by direct methods. However, the results of physical tests indicate that the structure of the hardened paste is altered.

The results of the physical tests show that for many cements the strengths can be increased and the contraction on drying or the expansion in water storage decreased by the use of larger additions of gypsum than are permitted by current specifications. In some instances in the present investigation the strengths were increased by as much as 20 to 50 per cent, and the contraction decreased by as much as 30 to 50 per cent. For cements of low $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ and low alkali content the strengths were not increased nor was the contraction decreased by larger additions of gypsum. The cements high in $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content regardless of their alkali content, or cements high in alkalis regardless of $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content, require the larger additions of gypsum. Gypsum could be added in larger amounts than is permitted by present specifications without danger of delayed expansion.

A Large Dragline Excavator.

A dragline excavator of exceptionally large capacity and suitable for stripping overburden from mineral deposits and similar operations where a high rate of superficial or deep excavation is required, is manufactured in the United States by the Bucyrus-Eirie Co. The machine is electrically operated and, with a boom 180 ft. long, can operate a bucket with a capacity of 25 cu. yd.; with a 250-ft. boom the maximum bucket capacity is 14 cu. yd.